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Effect of the surface condition on the deviation from Matthiessen's rule in thin copper samples

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We have studied the electrical resistivity $\rho(d,T)$ of high-quality single-crystalline copper whiskers in the temperature range 4.2—40 K with particular emphasis on the influence of the surface conditions on the surface-induced deviations from Matthiessen's rule. Roughening the initial microscopically smooth sample surface by chemical etching increases the residual resistivity $\rho(d,0)$, while the temperature-dependent part $\Delta\rho(d,T)$ is decreased. The latter is also found for the recently observed surface-induced T^2 contribution to the resistivity of copper whiskers. These results are interpreted with the size-effect theory of Sambles *et al.*, wherein an angle-dependent specularity parameter is employed. Additionally a comparison is made between the variation of the surface- and impurity-induced deviations from Matthiessen's rule in copper with the residual resistivity.

I. INTRODUCTION

There are many different causes known for the various deviations from Matthiessen's rule (DMR) observed for the low-temperature electrical resistivity of metals. A survey dealing with the case of dilute alloys and strained samples can be found in the review articles by Bass,[1] Cimberle *et al.*,[2] and Wiser.[3] In thin high-purity samples with sufficiently large electron mean free path l_e the surface scattering of the conduction electrons is another important source for DMR. These surface-induced deviations from Matthiessen's rule (SIDMR) are of particular interest since until now there is only little known about the interaction of the electrons with the metal surface (for a review of the theory of surface scattering, see Okulov and Ustinov[4]).

The earlier experimental and theoretical results on the surface-scattering contribution to the temperature-dependent part of the resistivity

$$\Delta\rho(d,T)=\rho(d,T)-\rho(d,0) \tag{1}$$

has been thoroughly reviewed by Bass.[1] It was found that the experimentally observed SIDMR in thin wires, which is defined as the difference between the temperature-dependent resistivity of a sample with thickness d and the bulk material

$$\Delta_s(d,T)=\Delta\rho(d,T)-\Delta\rho(\infty,T) \tag{2}$$

is larger than predicted by the size-effect theory of Dingle.[5] The simplified assumptions of his theory are a spherical Fermi surface, an isotropic relaxation time, and a constant specularity parameter p, which is simply the probability of an electron being specularly reflected from the sample surface. Recently the theory of the surface-scattering contribution to the resistivity of thin wires has been improved by Pavlov[6] and Sambles *et al.*[7] who took into account the dependence of the specularity parameter on the angle of incidence of electrons onto the surface. Sambles *et al.* especially employed the theory of surface scattering due to Soffer,[8] which involves an angle-dependent specularity parameter as a function of the surface roughness $H\equiv h/\lambda_e$ (h is the rms asperity height and λ_e is the Fermi wavelength). Using their results it was possible to explain the magnitude and the temperature dependence of the SIDMR recently studied in different metals.[9–12] There are two very interesting features of the surface-scattering corrections to $\Delta\rho(d,T)$.

(1) $\Delta_s(d,T)$ varies linearly with T^2 in a quite limited temperature range whereby the slope $A_s = d\Delta_s(d,T)/d(T^2)$ depends on the sample thickness.[12–15] Recently the aluminum thin-film data of Sambles and Elsom[15] and our own results on copper whiskers[15] have shown a $d^{-2/3}$ dependence of the coefficient A_s, as predicted theoretically by Sambles and Preist.[16]

(2) In the low-temperature limit, where $\Delta\rho(\infty,T)\ll\rho(\infty,0)$, the surface scattering simply enhances the temperature dependence of the bulk resistivity. For very thin samples ($d/l_e\ll1$) with smooth surfaces ($H\lesssim0.4$) the calculation of Sambles and Preist[16] yields a linear relation between $\Delta\rho(d,T)$ and $\rho(d,0)$. This has been proved experimentally by Thummes *et al.*[17] and has enabled us to determine the bulk temperature dependence of the resistivity of copper whiskers from experiments on thin samples. In addition, on the basis of this result, the contribution of electron-electron scattering to the resistivity has been deduced from measurements on copper whiskers at very low temperatures by Thummes and Kötzler.[18]

In this paper we at first want to show our experimental results on the effect of the surface conditions on the SIDMR in copper whiskers in the range 4.2—40 K. This includes an analysis of the diameter dependence of the surface-induced T^2 contribution to the resistivity. A discussion of the surface effects on the low-temperature ($T<4.2$ K) enhancement of the resistivity has been published elsewhere.[17] Further, a comparison is made between the variation of surface- and impurity-caused deviations from Matthiessen's rule with the residual resistivity. For this purpose we use the available experimental data on the impurity-induced DMR in copper.

II. EXPERIMENTAL DETAILS

The single-crystalline copper whiskers, grown by the reduction of copper iodide with hydrogen, were annealed under low pressure of oxygen to remove the effects of magnetic impurities (see, e.g., Thummes and Mende[19]). We selected whiskers with $\langle 111 \rangle$ growth axes exhibiting hexagonal cross sections which are well approximated by a circular wire geometry. The temperature dependence of the resistance was measured by a four-wire low-frequency (84 Hz) technique using lock-in detection to an uncertainty of 0.1%. After inductive compensation of the voltage drop at a fixed temperature a resolution of 10 ppm was achieved for samples with a resistance of about 50 $\mu\Omega$. The whiskers were mounted in a temperature-controlled chamber (± 20 mK) inserted into an evaporation cryostat. Absolute resistances at 4.2 and 293.2 K were measured by a dc method to an uncertainty of 0.08%. To convert from values of resistance to resistivity the shape factor $C=\rho/R$ of the samples was determined from the measured room-temperature resistance using $\rho(\infty, 293.2\ \mathrm{K}) = 1.679\ \mu\Omega\,\mathrm{cm}$ ($\lesssim 0.5\%$) deduced from published data for bulk high-purity copper.[20] The sample diameter $d=(4A/\pi)^{1/2}$, where A is the cross-sectional area, was determined to an uncertainty of 1 μm by means of a microscope. As discussed recently,[12] various sources of uncertainty in $\Delta_s(d,T)$ cause us to limit our analysis to $T<30$ K. The theoretical temperature dependence of $\Delta_s(d,T)$ is calculated from the theory of Sambles *et al.*[7] using the recently evaluated bulk values of $\rho(\infty, 4.2\ \mathrm{K})$ and $\Delta\rho(\infty, T)$ for the copper whiskers.[12] It was shown that the size-effect data at constant temperatures can be described using $\rho(\infty,0) \simeq \rho(\infty, 4.2\ \mathrm{K}) = 0.273 \pm 0.003\ n\Omega$ cm, $l_e(4.2\ \mathrm{K}) = 242 \pm 2\ \mu$m and the tabulated bulk temperature dependence of the resistivity.

III. RESULTS AND DISCUSSION

A plot of $\Delta_s(d,T)$ versus T for three typical whiskers with as-annealed surface conditions and after etching the sample surface is shown in Fig. 1. It is evident from this figure that the SIDMR decreases with increasing surface roughness which is most pronounced for the thinnest whisker. This result is in accordance with the size-effect theory of Sambles *et al.* The solid and dashed curves in Fig. 1 represent the result of a least-squares fit of our data to this theory in the range 6–30 K using the roughness parameter H as a fit parameter. The H values obtained and other important sample parameters are summarized in Table I. For both surface conditions the theory provides a close fit from about 15 to 40 K. At the present we have no explanation for the systematical deviations below 15 K, where our measurements yield larger $\Delta_s(d,T)$ values than the theory. But it may be important that these deviations increase with decreasing sample diameter and decreasing surface roughness. Furthermore, it is surprising that the H values deduced from the data and the effect of the surface treatment on H decrease with increasing sample size (see Table I). A thickness dependence of the roughness parameter deduced from the theory of Sambles *et al.* has already been reported and discussed by Boughton[10] in his study of the SIDMR in gallium single crystals and in our previous papers on the SIDMR in etched whiskers[12] and polycrystalline copper wires.[21] This clearly indicates that the size-effect theory and the model for angular-dependent scattering from rough surfaces has to be refined in order to obtain accurate information on the sample surface from size-effect data.

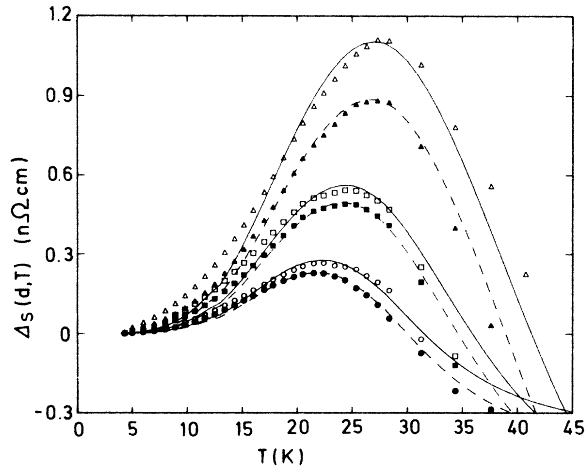

FIG. 1. Contribution $\Delta_s(d,T)$ of the surface scattering to the temperature-dependent part of the resistivity of the as-annealed, open symbols, and etched, solid symbols, samples 1: △,▲; 2: □,■; 4: ○,●. The solid and dashed curves are calculated from the theory of Sambles *et al.* using the H values which are listed with the sample data in Table I.

The existence of a surface-induced T^2 contribution to the resistivity of thin samples in a limited temperature range

$$\Delta_s(d,T) \sim A_s T^2 \tag{3}$$

is now well established but very little is known about the effect of the surface conditions on this term. Although we will not show the data, we have found experimentally that the temperature range where $\Delta_s(d,T)$ varies proportionally with T^2 is not changed after the surface treatment. Since the measured SIDMR is larger in whiskers with as-annealed surfaces our analysis of the T^2 term also yields larger A_s values for these samples which are compiled in Table I. The dependence of A_s on the sample thickness is illustrated in Fig. 2 where we have plotted A_s as a function of $[d/l_e(4.2\ \mathrm{K})]^{-1}$. The consideration of the residual electron mean free path allows us to include our recent experimental results on less pure copper wires [$l_e(4.2\ \mathrm{K}) = 71\ \mu$m] with etched surfaces (see Ref. 21). From Fig. 2 it is evident that there is no simple linear dependence of A_s upon $[d/l_e(4.2\ \mathrm{K})]^{-1}$ for both surface conditions. A least-squares analysis of the data yields

TABLE I. Sample parameters and data analysis. The surface conditions of the samples are denoted by "a," as-annealed, or "e," etched; the values of the surface roughness parameter H are deduced from a fit of the SIDMR to the theory (see Fig. 1); A_s is the slope of the T^2 term of the SIDMR. Bulk values: $\rho(\infty,4.2\ \mathrm{K})=0.273$ nΩ cm; $l_e(4.2\ \mathrm{K})=242\ \mu$m.

Sample	d (μm)	Surface condition	$\rho(d,4.2\ \mathrm{K})$ (nΩ cm)	H	A_s (pΩ cm K^{-2})
1	7.5	a	6.18	1.30±0.1	2.09±0.05
		e	9.28	1.69±0.1	1.84±0.05
2	14.5	a	3.17	0.90±0.1	1.35±0.05
		e	5.00	1.03±0.1	1.23±0.05
3	21.0	a	2.26	0.70±0.05	1.00±0.03
		e	4.09	0.96±0.05	0.81±0.03
4	26.6	a	1.68	0.64±0.05	0.76±0.03
		e	2.86	0.75±0.05	0.70±0.03
5	36.7	a	1.30	0.45±0.05	0.63±0.02
6	40.2	a	1.12	0.41±0.03	0.54±0.01
		e	1.97	0.49±0.03	0.53±0.01
7	54.5	a	0.88		0.41±0.01
8	59.1	a	0.86	0.30±0.03	0.40±0.01
		e	1.43	0.36±0.03	0.38±0.01
9	82.6	a	0.69	0.28±0.03	0.29±0.02
		e	1.03	0.32±0.03	0.27±0.02

$$A_s=-0.2\pm0.1+(0.24\pm0.02)[d/l_e(4.2\ \mathrm{K})]^{-0.66\pm0.05}\ \mathrm{p\Omega\,cm\,K^{-2}} \tag{4a}$$

for the as-annealed samples and

$$A_s=-0.09\pm0.08+(0.19\pm0.02)[d/l_e(4.2\ \mathrm{K})]^{-0.68\pm0.03}\ \mathrm{p\Omega\,cm\,K^{-2}} \tag{4b}$$

for the chemically etched samples including the wire data which is depicted by the dashed and solid curves, respectively, in Fig. 2. The same thickness dependence of A_s has been found by Sambles and Elsom[15] in their detailed reanalysis of aluminum thin-film data. A comparison of Eqs. (4a) and (4b) reveals that roughening the sample surface does not alter the thickness dependence of A_s. Only the slope decreases with increasing roughness. The slight difference in the intercept results from the fact that at the present there is no data available on as-annealed samples with $0.4\leq d/l_e(4.2\ \mathrm{K})\leq 1$. Recently we have experimentally confirmed the prediction of the theory of Sambles *et al.* that the SIDMR is negative in the whole temperature range for samples with $d/l_e(4.2\ \mathrm{K})>1$.[21] Therefore it is worthless to extrapolate the magnitude of the surface-induced T^2 contribution to larger sample thickness. The present result on the effects of the surface conditions on the T^2 dependence of the SIDMR supports our opinion that this T^2 term in copper is a consequence of the size effect combined with a T^5 dependence of the bulk resistivity.[12,21] This has already been suggested by Sambles and Preist.[16]

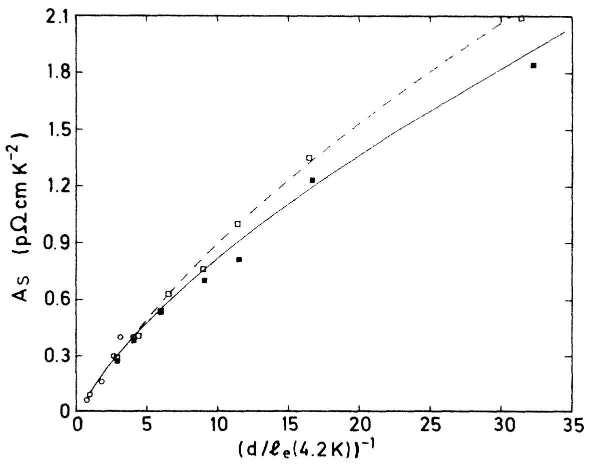

FIG. 2. Magnitude A_s of the surface-induced T^2 term for whiskers in the as-annealed, □, and etched state, ■. The open circles represent our recent results on etched polycrystalline copper wires (Ref. 21). The dashed and solid curve are calculated from Eqs. (4a) and (4b), respectively.

Since we now have available experimental results on the SIDMR in copper over a large range of the sample parameters it will be interesting to compare the residual resistivity dependency of the SIDMR with the impurity-induced DMR. For the published Al thin-foil data this has already been done by Nakamichi and Kino[22] and shown some remarkable differences. Unfortunately there is no recent comprehensive study of the impurity DMR in copper available and we have to rely on the earlier data. These have been reviewed by Cimberle *et al.*[2] and reexamined by Bergman *et al.*[23] Cimberle *et al.*[23] have shown that above a certain, experimentally definable residual resistivity the DMR at a fixed low temperature increases logarithmically with ρ(4.2 K). On the other hand the recent theoretical calculations of Bergman *et al.* reveal a more complex variation and excellent agreement with the experimental results on different metals.[3,23] However, we have considered both results and selected the data at 15.7 and 23.4 K for a comparison with the SIDMR. Supplementarily we include the results of Moussouros and Kos[24] on pure bulk copper, the size-effect data of Alderson and Hurd[25] on copper wires, and our own data on etched thin copper wires.[21] The various symbols in Fig. 3 represent the experimental data and the solid and dashed curves the empirical[2] and theoretical[23] dependence of the impurity-induced DMR in copper. It should be emphasized that in the case of the SIDMR data the residual resistivity ρ(4.2 K) is given by the sum of the bulk residual resistivity $\rho(\infty$, 4.2 K) and the additional size-effect contribution.

Let us first discuss briefly the bulk data. In Ref. 12 we have shown that the temperature dependence of the resistivity of the thickest whisker ($d = 142\ \mu$m) we have investigated is nearly equal to the bulk temperature dependence $\Delta\rho(\infty,T)$ of high-purity copper whiskers. This result is now supported by the excellent agreement between our bulk values and the empirical, respectively, theoretical limits for $\Delta\rho(T)$ at 15.7 and 23.4 K (see Fig. 3). Additionally, it turns out that the whisker data is nearly equal to the result of Moussouros and Kos[24] on less pure bulk copper samples.

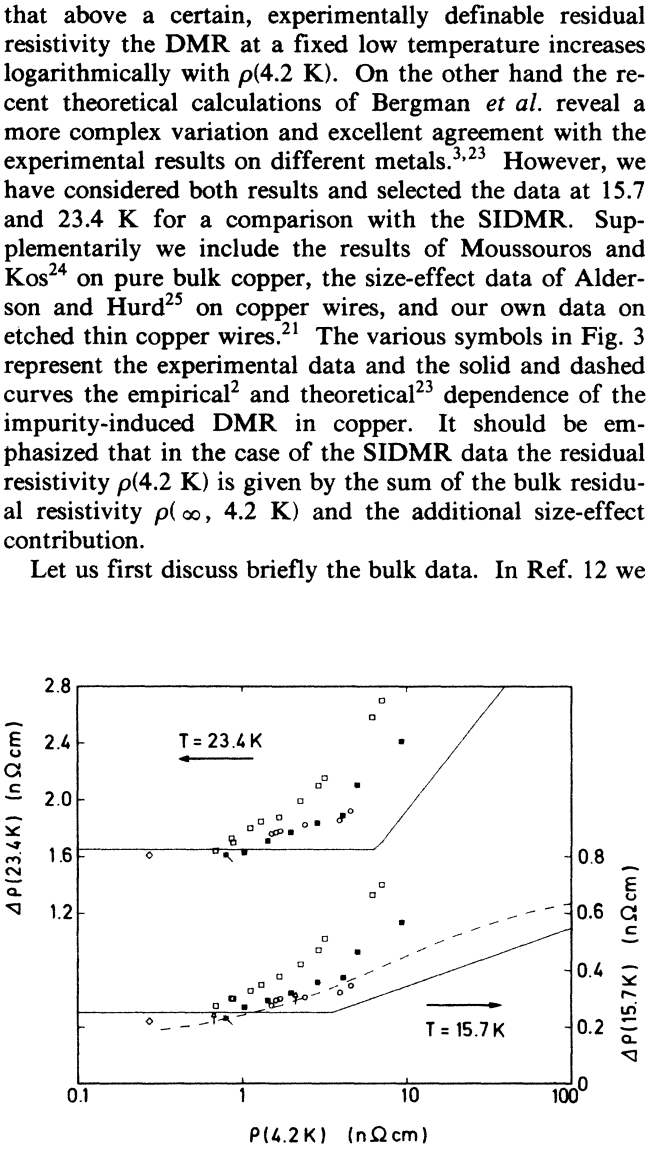

FIG. 3. Temperature-dependent part of the resistivity $\Delta\rho(T)$ of copper as a function of the residual resistivity ρ(4.2 K) at 15.7 and 23.4 K. *SIDMR data*: □, as-annealed whiskers; ■, etched whiskers; ○, etched wires (see Ref. 21); ⍋, wire data of Alderson and Hurd (Ref. 25). *DMR data*: ——, empirical results of Cimberle *et al.* (Ref. 2); — — —, theoretical calculations of Bergman *et al.* (Ref. 23) (their Fig. 2). *Bulk data*: ◇ our deduced whisker data (Ref. 12); ✱, wire data of Moussouros and Kos (Ref. 24).

It is evident from Fig. 3 that the magnitude of the SIDMR deviates systematically from the variation of the impurity-caused DMR with the residual resistivity. The ρ(4.2 K) dependence is obviously stronger than the log[ρ (4.2 K)] slope of the DMR deduced by Cimberle *et al.*[2] On the other hand we observe a qualitative agreement between the SIDMR data on etched whiskers and wires at 15.7 K and the DMR calculations of Bergmann *et al.*[23] up to about ρ(4.2 K) = 5 nΩ cm (see the dashed curve in Fig. 3) but positive deviations for still finer samples. The difference between SIDMR and impurity DMR is most pronounced for the as-annealed whiskers with smooth surfaces. At the same value of ρ(4.2 K) these samples show at least twice as large SIDMR as the etched samples. Note that there is no indication for a saturation of the surface-induced DMR for very thin samples [i.e., large values of ρ(4.2 K)]. A closer inspection of Fig. 3 reveals some small differences between the ρ(4.2 K) dependence of the SIDMR data of whiskers and wires. These are explained by the different bulk residual resistivities of these samples. For the copper wires we have found $\rho(\infty$,4.2 K) = 0.95 nΩ cm which is about a factor 3.5 larger than the value deduced from the size-effect data of the whiskers (see Table I and Ref. 12). Since the magnitude of the size effect in a thin sample is given by the ratio $\rho/\rho_\infty = \rho(d,4.2\ \mathrm{k})/\rho(\infty,4.2\ \mathrm{K})$, the SIDMR of samples with different bulk residual resistivity but equal values of ρ/ρ_∞ should be comparable. The attempt to quantify the variation of our data yields a $(\rho/\rho_\infty)^{2/3}$ dependence of the SIDMR in the range 10–20 K. This is illustrated in Fig. 4 where we have plotted $\Delta_s(d,T)$ as a function of $(\rho/\rho_\infty)^{2/3}$ at 12 and 20 K. In this plot the agreement between the variation of the magnitude of the SIDMR in etched whiskers and wires is much better than in Fig. 3. Moreover it is obvious that the data of the thicker wires with $(\rho/\rho_\infty)^{2/3} \lesssim 2$, including the former result of Alderson and Hurd[25] at 12 K, supplement those obtained from the etched whiskers. Unfortunately at the present we have no data for very small values of ρ/ρ_∞ to investigate the lower limit of the $(\rho/\rho_\infty)^{2/3}$ dependence of the SIDMR in copper at a fixed temperature. A comparison between the present experimental result on the residual resistivity dependence of the SIDMR with the size-effect theory of Sambles *et al.* is not very meaningful since, as shown above (see Table I), the roughness parameter deduced from this theory varies systematically with sample size. However, it is interesting to note that the theoretical calculations of Boughton and Neighbor[26] have shown an influence of the Fermi-surface geometry on the SIDMR. Assuming totally "diffuse" scattering of electrons at the samples surface (which corresponds to $H \gg 1$ in the

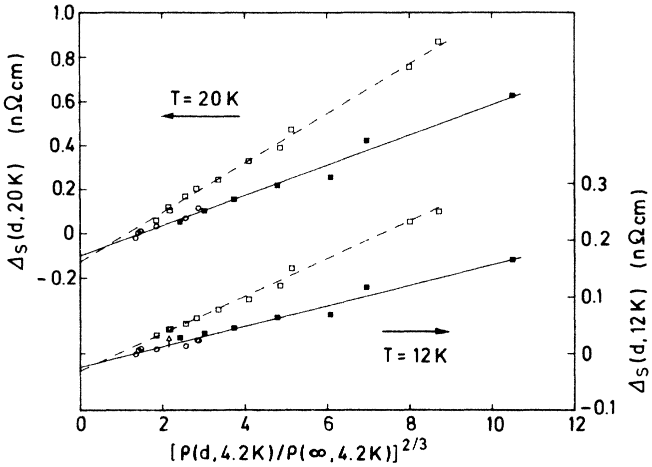

FIG. 4. Surface-scattering contribution $\Delta_s(d,T)$ to the temperature-dependent part of the resistivity as a function of $[\rho(d,4.2\text{ K})/\rho(\infty,4.2\text{ K})]^{2/3}$ at 12 and 20 K. □, as-annealed whiskers; ■, etched whiskers; ○ etched wires [Kuckhermann *et al.* (Ref. 21)]; ♀, wire data of Alderson and Hurd (Ref. 25) at 12 K. The dashed and solid straight lines show the results of a linear regression of the data.

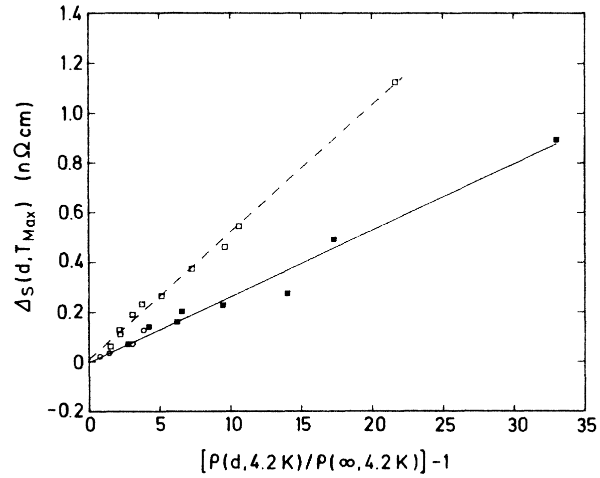

FIG. 5. Plot of the variation of the maximum value of the SIDMR $\Delta_s(d,T_{\max})$ with $[\rho,(d,4.2\text{ K})/\rho(\infty,4.2\text{ K})]-1$. □, as-annealed whiskers; ■, etched whiskers; ○, etched wires (Ref. 21). The dashed and solid straight lines represent fits to a linear dependence on $\rho/\rho_\infty-1$.

theory of Sambles *et al.*) and $d/l_e \lesssim 0.1$, the authors found that: (1) $\Delta_s(d,T)$ increases logarithmically with $\rho(4.2\text{ K})$ for a spherical Fermi surface; (2) $\Delta_s(d,T)$ increases linearly with $\rho(4.2\text{ K})$ for a cylindrical Fermi surface. Our experimentally deduced $(\rho/\rho_\infty)^{2/3}$ dependence of the SIDMR in copper is intermediate between these two distinct types of behavior.

A further qualitative difference between SIDMR and impurity-induced DMR follows from the comparison of the data at the temperature $T_{\max}$ where the maximum occurs. First of all, in the case of surface scattering $T_{\max}\approx 20$—30 K, where $d=l_e(T_{\max})$, is smaller than for impurity scattering where $T_{\max}\approx 60$ K (see, e.g., the review article by Bass[1]). According to Cimberle *et al.*[2] the DMR in copper still varies linearly with the logarithm of $\rho(4.2\text{ K})$ at this temperature similar to the dependence observed at lower temperatures (see Fig. 3). On the contrary, our SIDMR data show a behavior which differs from the variation of $\Delta_s(d,T)$ at lower temperatures displayed in Fig. 4. The analysis of the data reveals a linear dependence of the maximum of the SIDMR $\Delta_s(d,T_{\max})$ on the residual resistivity as can be seen in Fig. 5. Taking into account the different values of $\rho(\infty,4.2\text{ K})$, we again find overall agreement between the experimental results on etched wires and whiskers. As already shown above, the SIDMR is clearly reduced after etching the sample surface but the linear relation between $\Delta_s(d,T_{\max})$ and $\rho(d,4.2\text{ K})/\rho(\infty,4.2\text{ K})$ is found for both surface conditions. The surface treatment decreases the slope of the regression lines by about a factor 2, whereas the intercept does not depend on the surface state and is approximately zero. Without showing the result we note that a calculation from the theory of Sambles *et al.*[7] using a constant roughness parameter yields a nearly quadratic variation of $\Delta_s(d,T_{\max})$ with $\rho(d,4.2\text{ K})$.

IV. SUMMARY AND CONCLUSIONS

We have shown that the surface conditions have a significant influence on the deviations from Matthiessen's rule induced by the scattering of the conduction electrons at the sample surface. The magnitude of the SIDMR in copper whiskers is found to decrease after roughening the surface by chemical etching. Our results on the temperature dependence of the SIDMR are in quantitative agreement with the size-effect calculations of Sambles *et al.*[7] although it turns out that the surface roughness parameter decreases with increasing sample size. For both surface states, i.e., for as-annealed and etched samples, a surface-induced T^2 contribution to the resistivity has been identified in a limited temperature range. The $d^{-2/3}$ dependence deduced for the thickness dependence of the coefficient A_s in the term $\Delta_s(d,T)\sim A_sT^2$ is independent of the surface condition.

It was shown that the dependence of the SIDMR on the residual resistivity differs from the variation reported for the impurity-induced DMR in copper. The magnitude of the SIDMR at a fixed low temperature increases stronger than the empirical $\log[\rho(4.2\text{ K})]$ dependence deduced by Cimberle *et al.*[2] for impurity scattering. This deviation is most pronounced for the as-annealed whiskers with smooth surfaces. Between 10 and 20 K the SIDMR data at a fixed temperature can be described by a $[\rho(4.2\text{ K})]^{2/3}$ dependence, whereas the maximum of the SIDMR, which occurs at $T_{\max}\approx 20$—30 K, increases linearly with the residual resistivity. These results cannot be explained by the size-effect theory assuming a constant surface roughness. But in agreement with the theory we have confirmed that the SIDMR is comparable in copper samples with different residual resistivities, e.g., different purity, but equal ρ/ρ_∞ values and surface conditions.

We plan to pursue the study of the SIDMR in fine copper samples with improved purity and surface conditions and hope that the results will provide more details about the surface scattering of the electrons and hints for a refinement of size-effect theory to explain the experimental results.

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